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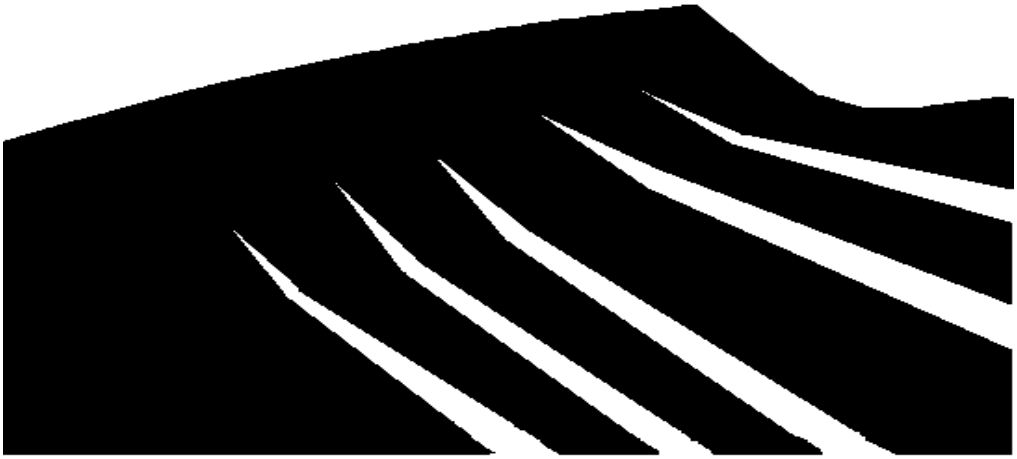
SOLID ROCK COLUMN EXPERIMENT

LOS ALAMOS

QUALITY PROGRAM



APPROVAL FOR RELEASE

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I. R. TRIAY - PRINCIPAL INVESTIGATOR Signature on file	DATE Date on file
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Los Alamos
Yucca Mountain Site Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	01/08/87	N/A	Not applicable
R1	03/31/87	N/A	History of Revisions not required until Rev. 3.
R2	03/13/89	N/A	History of Revisions not required until Rev. 3.
R3	08/13/92	All	Complete rewrite. Revisions 0-2 of this procedure were previously identified as TWS-INC-DP-61. Revision 3 was identified as LANL-INC-DP-61.
R4	03/21/97	All	Revised to comply with LANL-YMP-QP-06.3 requirements.

SOLID ROCK COLUMN EXPERIMENT

1.0 PURPOSE

The purpose of this detailed technical procedure (DP) is to study the dynamic transport tests on solid rock columns by studying the flow of known tracer spikes with a known flow rate through the column for the Yucca Mountain Site Characterization Project (YMP).

2.0 SCOPE

This procedure applies to all YMP personnel who perform solid rock experiments as part of the Dynamic Transport Task of the Los Alamos National Laboratory (Los Alamos) YMP.

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Procedure for Documenting Scientific Investigations
LANL-YMP-QP-08.1, Identification and Control of Samples
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-YMP-QP-17.6, Records Management
LANL-CST-DP-35, pH Measurement
LANL-CST-DP-60, Preparation of NTS Samples for LANL YMP Solid Core Experiments
CST-SOP-37, Rules for Handling Radioactive Material at TA-48
Rheodine 9125 Injection Valve instruction sheet (or instruction sheet of equivalent)
ISCO LC-2600 Syringe Pump user's manual

4.0 DEFINITIONS

4.1 Tracer solution

The tracer solution is the solution to be injected into the column containing the substance whose transport behavior is to be studies.

4.2 Eluate

The eluate is the solution resulting from an elution process.

4.3 Collection time

The collection time is the time interval between collection of eluate samples.

5.0 RESPONSIBILITIES

- Principal Investigator (PI)
- Procedure Users

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or the current work practice is documented in accordance with QP-03.5, subsection 6.1.6.

- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.

- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

6.1 Principle

This DP is utilized by forcing a ground water solution that contains a radioactive tracer through a column of solid rock. The amount of tracer that sorbs on the rock can be calculated by measuring the concentrations of the tracer in the initial solution and the solution that has been pushed through the rock.

6.2 Equipment and Hardware/Software

Equipment needed to conduct this DP is listed below. Items equivalent to those listed below may be used provided they perform the same function with an acceptable level of performance as judged by the user or the PI.

- high pressure syringe pump
- injection valve
- commercially available hand tools (micrometer, caliper, ruler)
- machine shop tools: lathe, drill press, rock polisher, rock saw
- vacuum oven
- calibrated balance

- fraction collector
- pH meter
- high pressure tubing

Materials and supplies include: Brass threaded tube with stainless steel end caps, O ring seals (see Attachment 1), polyether etherketone (PEEK) capillary tubing, teflon tubing, and vials.

6.2.1 Equipment Malfunctions

The equipment needed to conduct this experiment must be clean and in good repair.

6.2.2 Safety Considerations

Ensure compliance with CST Division Environmental Safety and Health Operational Policy Statement.

6.2.3 Equipment and Materials

Tracer solution for these experiments are often radioactive and should be handled in accordance with Rules for Handling Radioactive Material at TA-48 (CST-SOP-37). Injection of radionuclides often leaves residue sorped to minerals comprising the solid rock column. The column after injection should be handled as potentially contaminated and if to be disposed, treated as low level waste.

6.3 Preparatory Verification

6.3.1 Hold Points

There are no hold points for this procedure.

6.3.2 Calibration

6.3.2.1 Balances used for weighing are required to calibrated pursuant to QP-12.3. When data are collected from a balance, the unique identifier number of that balance is recorded in the user's laboratory notebook along with the data collected.

6.3.2.2 The pH meter used for any pH determinations shall be used in accordance with DP-35. The unique identifier of the pH meter shall be recorded in the user's laboratory notebook.

6.3.2.3 The caliper and micrometer used are commercial grade instruments that do not require calibration pursuant to QP-12.3.

6.3.3 Environmental Conditions

No special environmental conditions are required for this DP. If any special conditions are used, they will be recorded according to subsection 6.7 of this DP.

6.4 Control of Samples

The unique identifier of the solid rock core will be carried from LANL-CST-DP-60. If more than one solid rock core has been prepared from the same NTS sample an additional alphanumeric character will be added to the identifier in order to maintain uniqueness. Sample identification and control are governed by QP-08.1.

6.5 Implementing Procedure

6.5.1 Preparation of rock column for the experiment

6.5.1.1 Select sample from NTS core material. If more than one column is to be prepared from the same NTS sample, an additional alphanumeric character will be added to the identifier in order to maintain uniqueness.

6.5.1.2 Cut the core to the desired length with a low speed diamond saw to make the wafer.

6.5.1.3 Core the sample with the desired diameter diamond core bit using the coring machine.

6.5.1.4 Measure the diameter of the core to be used in the column to within 0.05 mm using the calipers or micrometer.

6.5.1.5 Encapsulate the core with epoxy inside stainless steel or brass tubing.

6.5.1.6 Smooth both ends using isomet polisher or lathe with diamond bit.

6.5.1.7 Measure the length of the encapsulated column to within 0.05 mm, using caliper or micrometer.

6.5.2 Determination of saturated weight of tuff

6.5.2.1 Determine the dry weight of a representative piece by drying an adjacent piece of the tuff core in a vacuum oven at 30° - 40° C until a constant weight is obtained. Weigh the piece weekly until the weight stabilizes within 0.01 g.

6.5.2.2 Determine the saturated (wet) weight of the piece by placing the piece in a small beaker of deionized water. Place the beaker in a vacuum oven at room temperature. Weigh the piece weekly until the weight stabilizes within 0.01 g and record the wet weight in the notebook.

6.5.2.3 Measure the diamond and length of the representative piece and record the data in the notebook.

6.5.3 Experiment preparation

6.5.3.1 Equilibrate the column by placing it in a beaker of the appropriate ground water (specified by the PI). Place the beaker in a vacuum oven at room temperature. Weigh weekly until a constant weight is obtained within 0.01 g. Check the pH of the equilibrating water periodically according to DP-35. After the column is saturated, record the final pH of the solution in the notebook.

6.5.3.2 Assemble the column (refer to Attachment 1 for detail).

6.5.3.3 Connect the confining vessel to the pump and the collector by capillary PEEK tubing (see Attachment 1).

6.5.3.4 Establish the desired flow rate through the solid rock column according to the user's manual for the pump and visually inspect system for leakage.

6.5.3.5 Prepare a solution of the appropriate groundwater containing the tracer(s) to be used. The source of the groundwater and tracer(s) are specified by the PI. Record the type of tracer and the method of preparation in the notebook.

6.5.3.6 Measure the pH of the solution according to DP-35 and record in the notebook.

6.5.3.7 Place an aliquot of the tracer solution in a capped container (the tracer concentration of this aliquot will be used to determine the initial concentration of the tracer) and record the volume in the notebook.

6.5.3.8 Weigh the vials to be used for eluate collection to within 0.001 g and position them in the fraction collector or on the balance.

6.5.3.9 Record collection time.

6.5.3.10 Load the tracer spike into the sample loop of the injection valve according to the instruction sheet of the injection valve.

6.5.3.11 Ensure that the following data are recorded in the laboratory notebook before starting the experiment:

- Type of column material (hole and depth) and unique sample ID (SMF#) of the sample used to prepare the column (carried from the manipulation procedure DP-60). If more than one column has been prepared from the same NTS sample, an additional alphanumeric character will be added to the identifier in order to maintain uniqueness.
- Unique identifier of the balance.
- The diameter and the length of the solid rock column.
- The groundwater used.
- The tracer used and the method of preparation.
- The pH of the solution.
- The dried weight and saturated weight of the representative piece.
- The operating pressure of the pump required to maintain the desired flow fate through the solid rock column.
- The volume of the aliquot of tracer solution.
- Tare weight of the collection vials.
- Collection time.

6.5.4 Conducting the experiment

6.5.4.1 Inject spike and record the injection time. The time consists of the date and the 24-hour clock time.

6.5.4.2 Start sample collection (either start the fraction collector or zero the balance used in collection).

6.5.4.3 Record start time for eluate collection for each vial when using a balance in the collection process. The time consists of the date and the 24-hour clock time.

6.5.4.4 Weigh each vial after eluate collection to within 0.001 g.

6.5.4.5 Determine the amount of tracer in the eluate using the appropriate analytical method.

6.5.4.6 Ensure that the following data are recorded in the notebook:

- Injection time.
- Start time of eluate collection for each vial when using a balance in the collection process.
- Weight of each vial after eluate collection.
- Collection time.
- Type of analytical instrument used for eluate analysis.
- Pump pressure reading interval (PI discretion)

6.5.5 Upon completion of the experiment, verify that no radical pump pressure changes have taken place since the beginning of the experiment. Verify that the information specified in subsection 6.5.3.11 and 6.5.4.6 have been recorded in the laboratory notebook.

6.6 Data Acquisition and Reduction

6.6.1 The data will be recorded in the user's laboratory notebook. Users should verify that data have been recorded properly.

6.6.2 The tare and final weight of each of the eluate vials are recorded. These data are later reduced by subtracting the tare weights from final weights of the collection vials in order to obtain the amount of eluate collected. The absolute or relative concentration of the tracer in the eluates is recorded. The relative concentration refers to amount of tracer in the eluate compared to the initial amount of tracer in the spike.

6.7 Potential Sources of Error and Uncertainty

A potential source of error can be evaporation of the eluate in the collection vials prior to the determination of the final weights of the collection vials. Keep the collection vials capped after eluate collection.

The most common sources of error that result in data rejection are leakage or plugging of the column. Leakage can be caused by cracking of the solid rock sample in the column, a crack in the epoxy encapsulation or failure of the O rings to maintain the seal. Radical changes in the operating pressure of the pump are indicative of leakage.

7.0 RECORDS

Records generated as a result of this DP are entries in laboratory notebooks or attachments to laboratory notebooks. The documentation should consist of any applicable items identified in Section 6.0 of this procedure. Laboratory notebooks should be kept in accordance with QP-03.5.

All records should be submitted to the Records Processing Center in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

Proper recording of data specified in subsections 6.5.3.11 and 6.5.4.6 constitute the acceptance criteria for this DP.

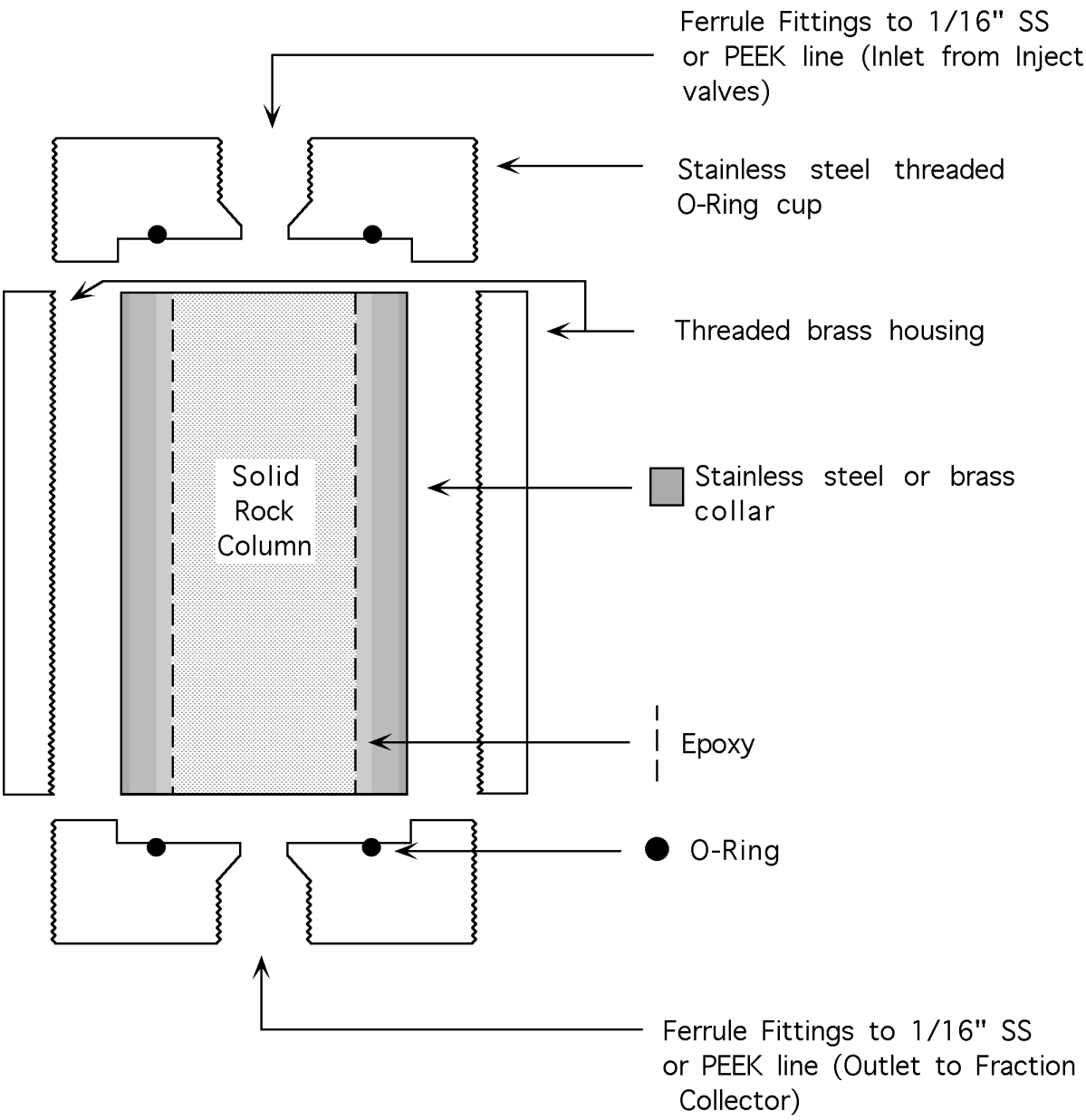
9.0 TRAINING

The PI or his designee will train the investigator assigned to use this DP. The personnel assigned to this work will be qualified by formal training through observation and evaluation of the performance of the trainee as he follows this DP. Training is documented according to QP-02.7.

10.0 ATTACHMENTS

Attachment 1: Cross Sectional View of Solid Rock Column (1 page).

Cross Sectional View of Solid Rock Column



(Not to Scale)